Effect of thickness on elastic-property measurements of thin films using atomic force acoustic microscopy

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Overview

•Basics of AFAM

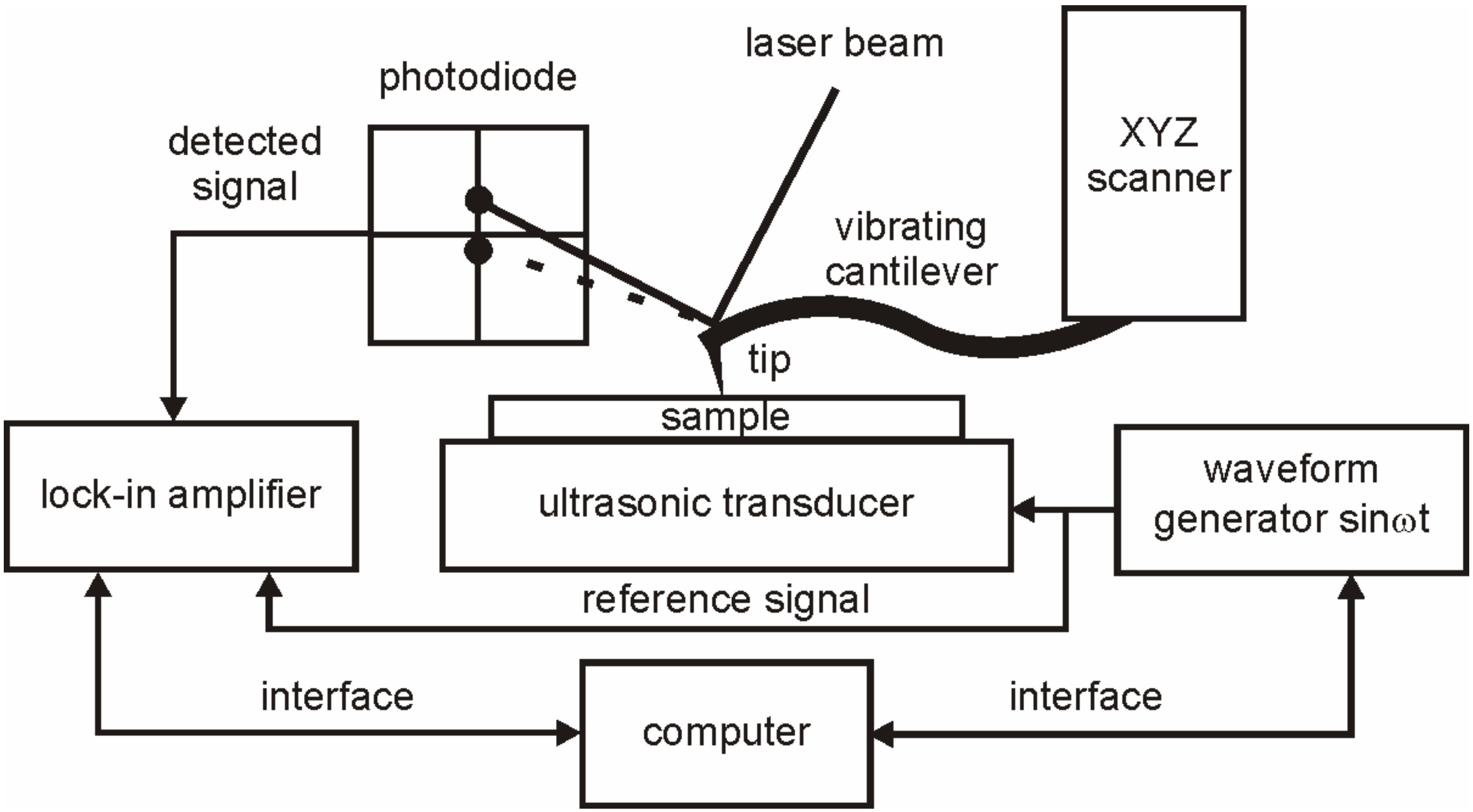
•Characterization of the investigated thin-film samples; measurements of thickness, microstructure and texture

•Experimental results

•Stress field in AFAM experiments.
When can we neglect the influence of a substrate?

•Conclusions

Experimental set-up



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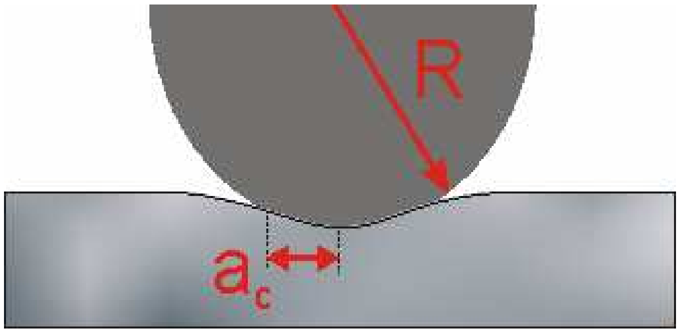
Experimental procedure

cantilever:
k_c = 44 N/m
f_1 = 168 kHz
f_2 = 1042 kHz

applied force:
880 nN – 2640 nN,
measured: f_{1con}, f_{2con},
calculated: k^*

calibration:
reference sample: single-crystal nickel
orientation: <100>
indentation modulus: $M_{<100>}$ = 219 ± 2 GPa



$$E^*_{sam} = E^*_{ref}\left(\frac{k^*_{sam}}{k^*_{ref}}\right)^n \quad \begin{cases} n = 1 \text{ for a flat punch} \\ n = 3/2 \text{ for a hemisphere} \end{cases} \quad \frac{1}{E^*} = \frac{1}{M_{tip}} + \frac{1}{M_{sample}}$$

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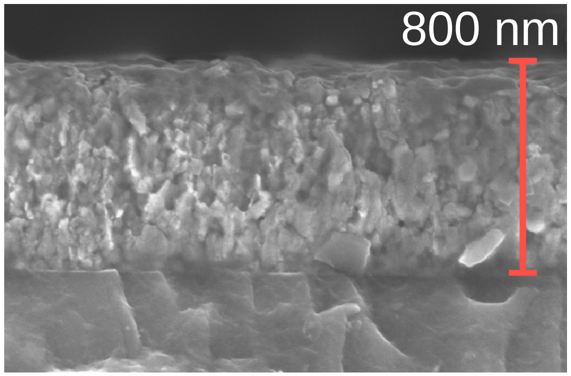
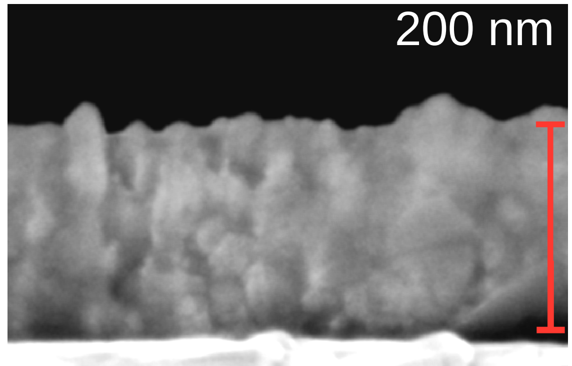
Cross-sectional SEM images of thin-film nickel samples

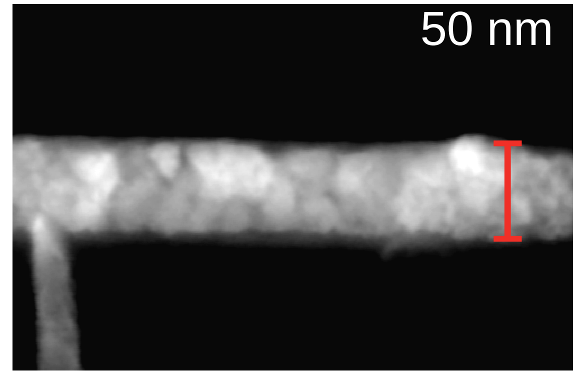
$Ni_{800\ nm}$



$Ni_{200\ nm}$



$Ni_{50\ nm}$



Measured thickness:

772 ± 5 nm

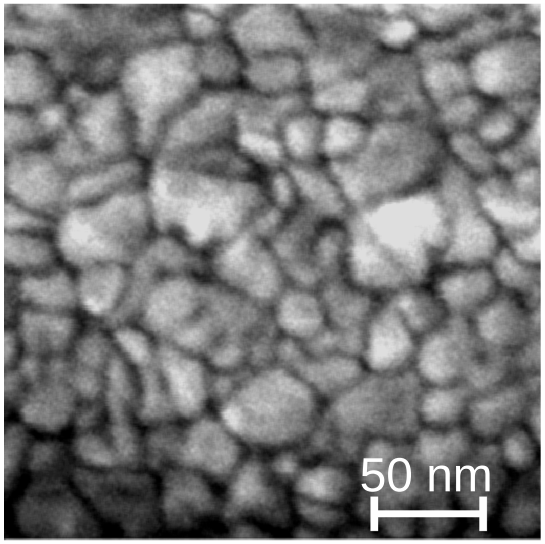
204 ± 4 nm

53 ± 2 nm

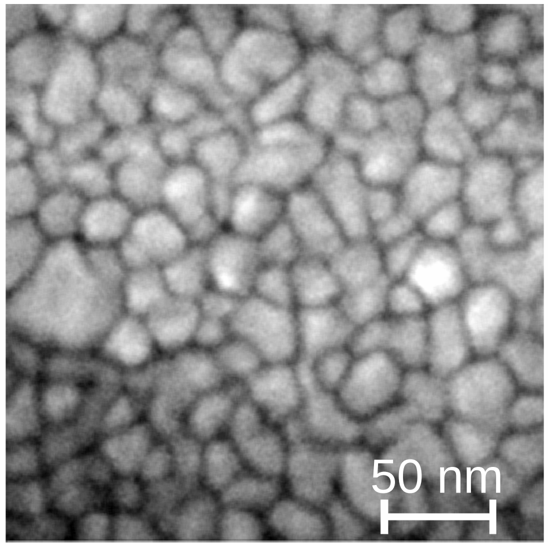
Method: DC magnetron sputtering
Substrate: <100> silicon
Preparation: W.H. Rippard and S.E. Russek, NIST Boulder

Plane view SEM images of thin-film nickel samples

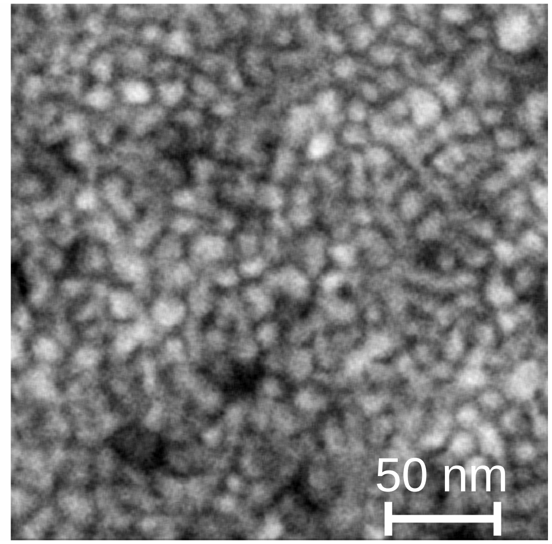
$Ni_{800\ nm}$



$Ni_{200\ nm}$



$Ni_{50\ nm}$



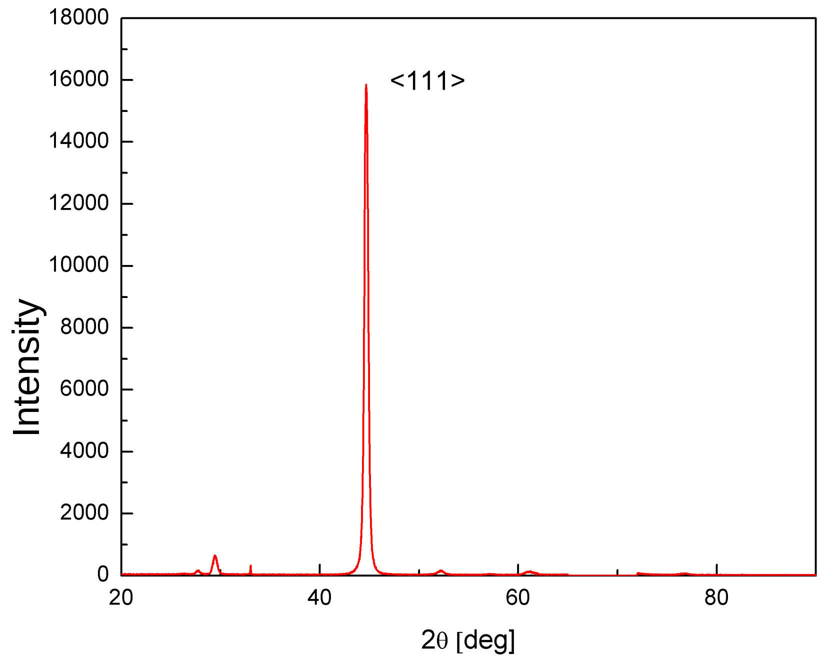
	$Ni_{800\ nm}$	$Ni_{200\ nm}$	$Ni_{50\ nm}$
Average grain diameter d	27 ± 10 nm	22 ± 5 nm	10 ± 3 nm
Surface roughness R_q	1.2 nm	0.6 nm	0.5 nm

Texture study for 800 nm film

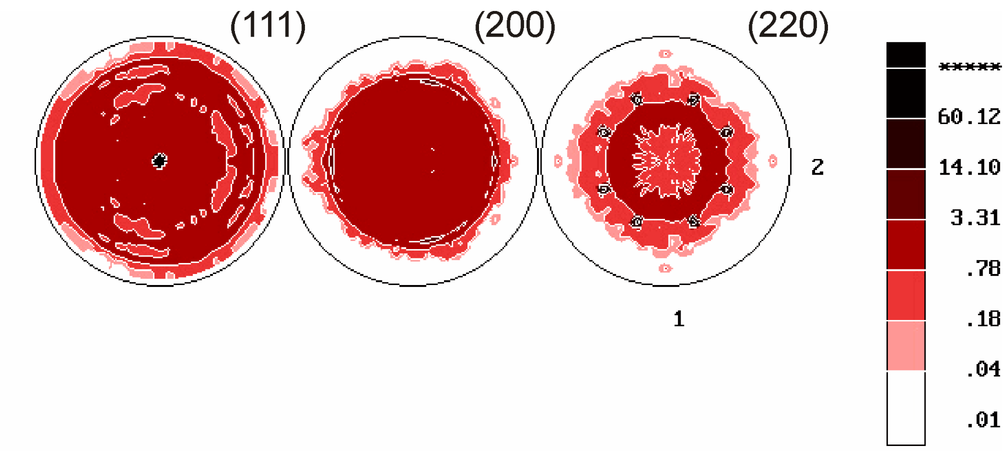
Ni is elastically anisotropic

Literature values for single-crystal Ni $M_{<100>}$= 220 GPa $M_{<111>}$= 250 GPa

XRD spectrum



Pole figures



Literature value for <111> textured material **M = 242 ± 10 GPa**

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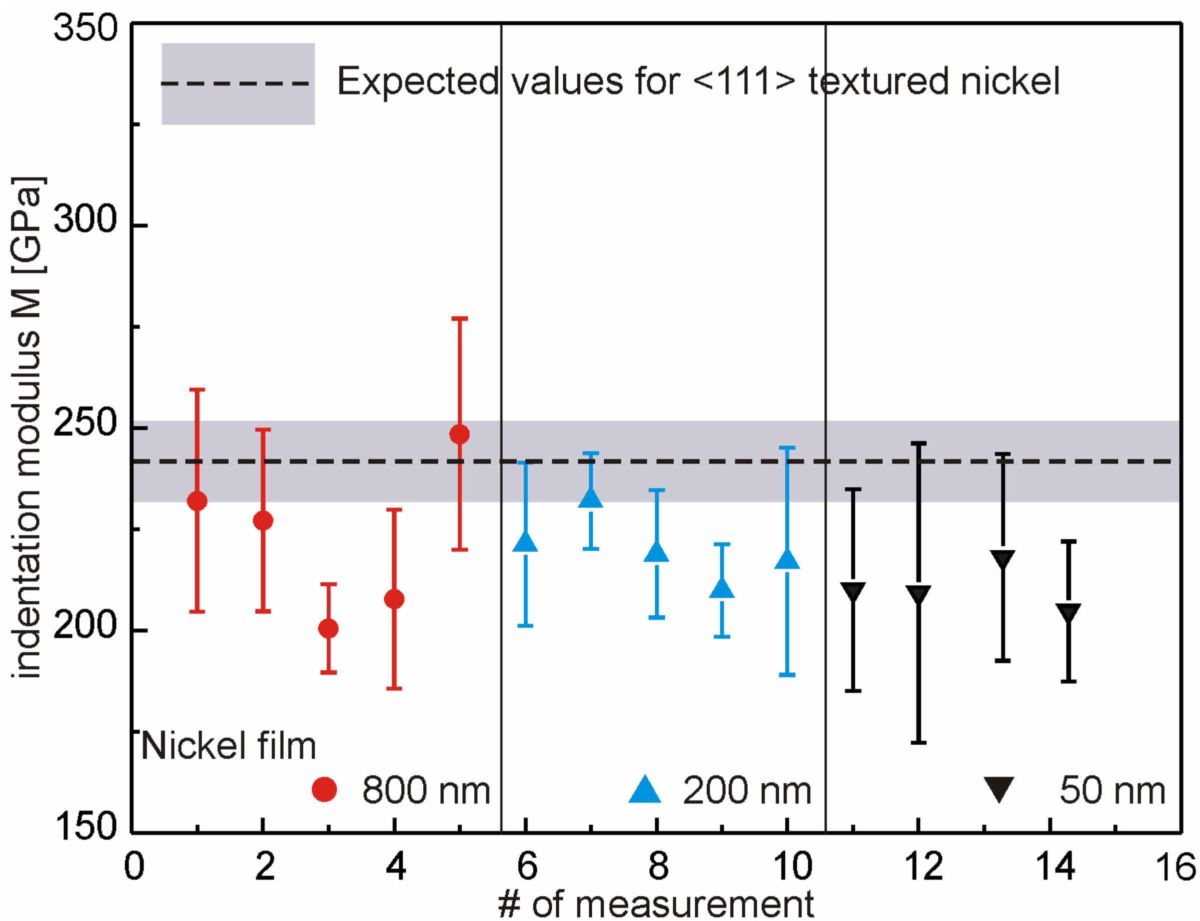
AFAM results for indentation modulus M



Average values of M

Ni_{800nm} 223 ± 28 GPa

Ni_{200nm} 220 ± 19 GPa

Ni_{50nm} 210 ± 26 GPa

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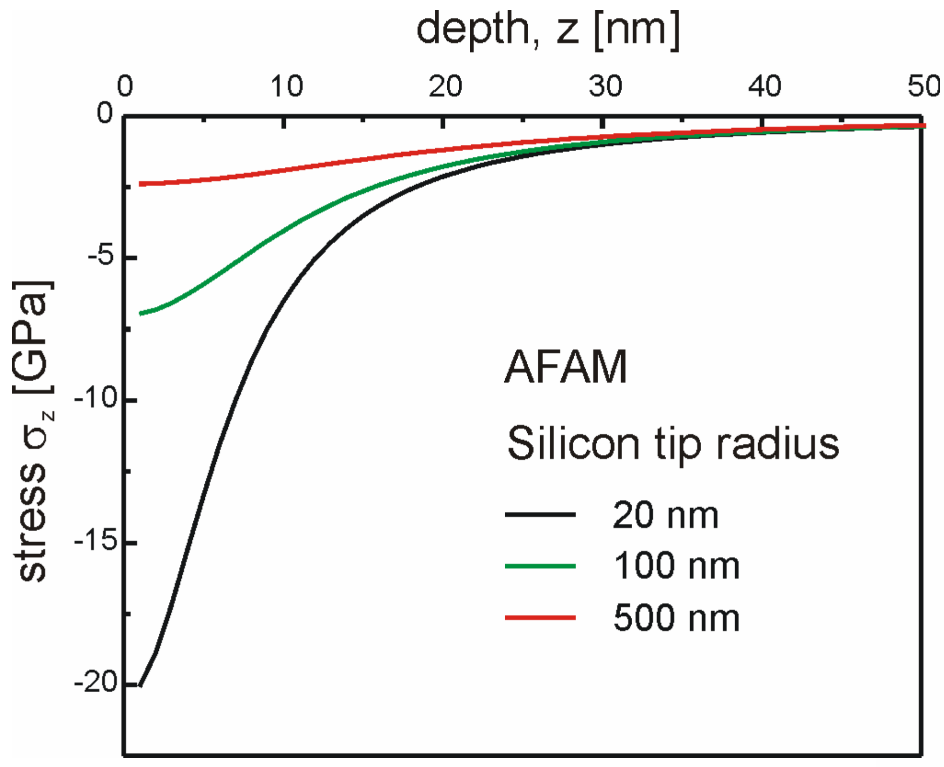
What can cause a reduction in values of elastic modulus?

- Large deformation of substrate at the interface of thin-film sample?

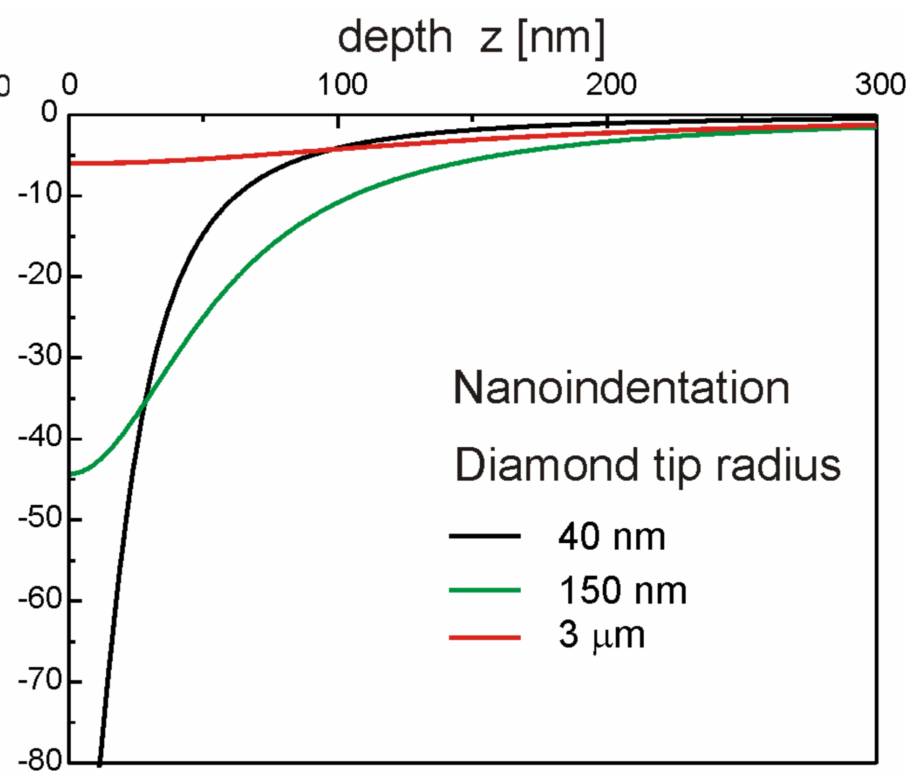
- Presence of closed pores (voids) and elemental impurities in the film?

- Change of elastic properties due to high intercrystalline volume content?

Stress field in AFAM and nanoindentation



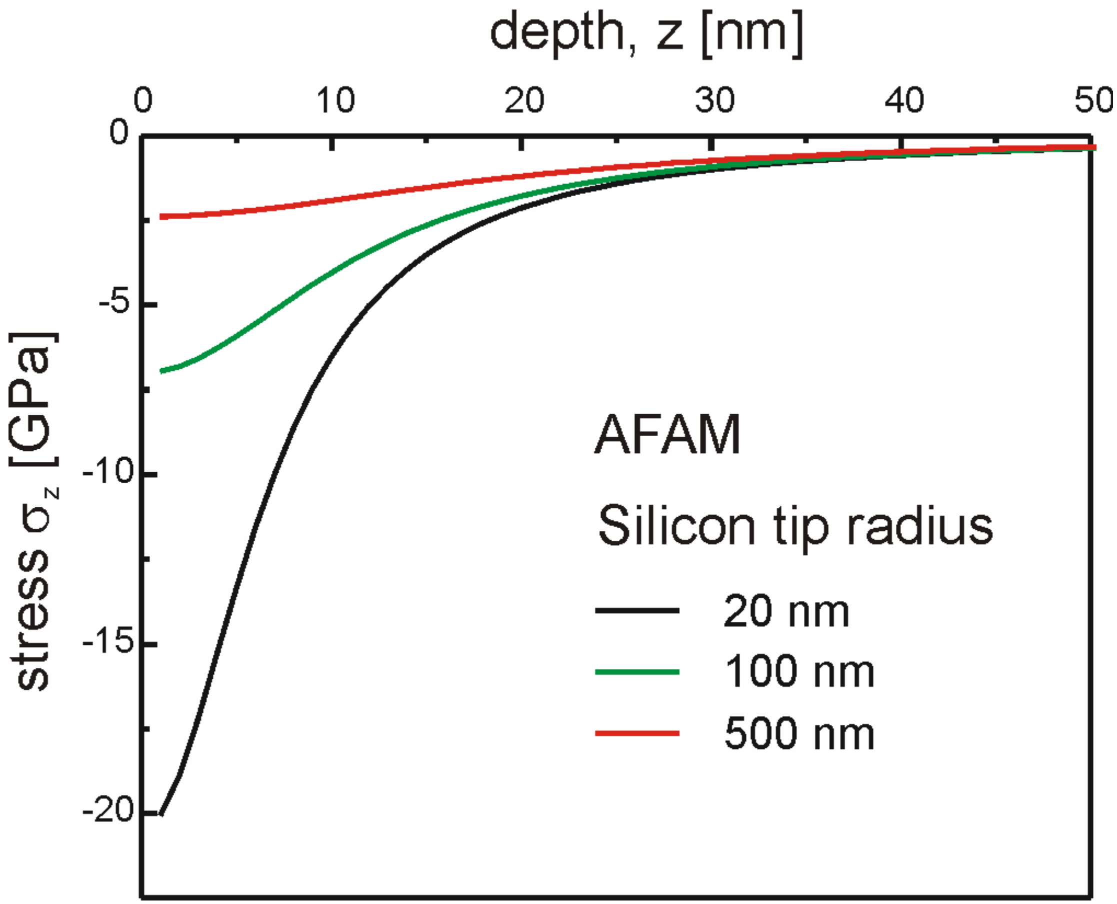
$F_c = 2\ \mu N$



$F_c = 300\ \mu N$

Stress fields in AFAM are smaller and decrease faster than in nanoindentation

Substrate influence



Stress field under a spherical indenter:

$$\sigma_z = -p_{\max}\left(1+\frac{z^2}{a_c^2}\right)^{-1}$$

$$p_{\max} = \frac{3F_C}{2\pi a_c^2}$$

$$a_c = \sqrt[3]{\frac{3RF_C}{4E^*}}$$

Deformation δ caused by the stress:

$$\delta = -\sigma_z \frac{\pi a_c}{2E^*}$$

Deformation of the substrate at the interface

R: 40 nm – 110 nm, max F_c = 2640 nN

δ_s - deformation at the surface

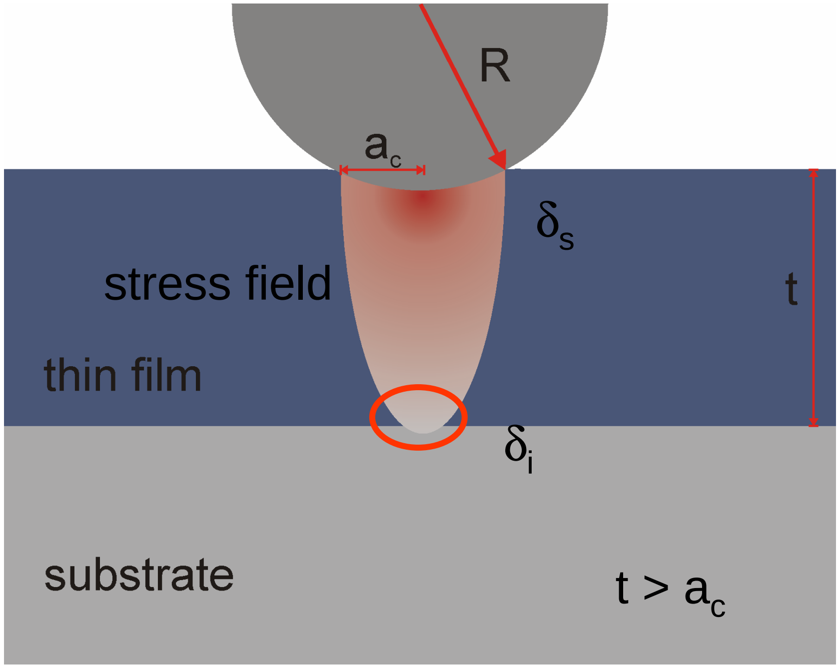
δ_i - deformation at the interface

We can neglect the substrate influence if

$$\delta_s \gg \delta_i$$

We estimated that:

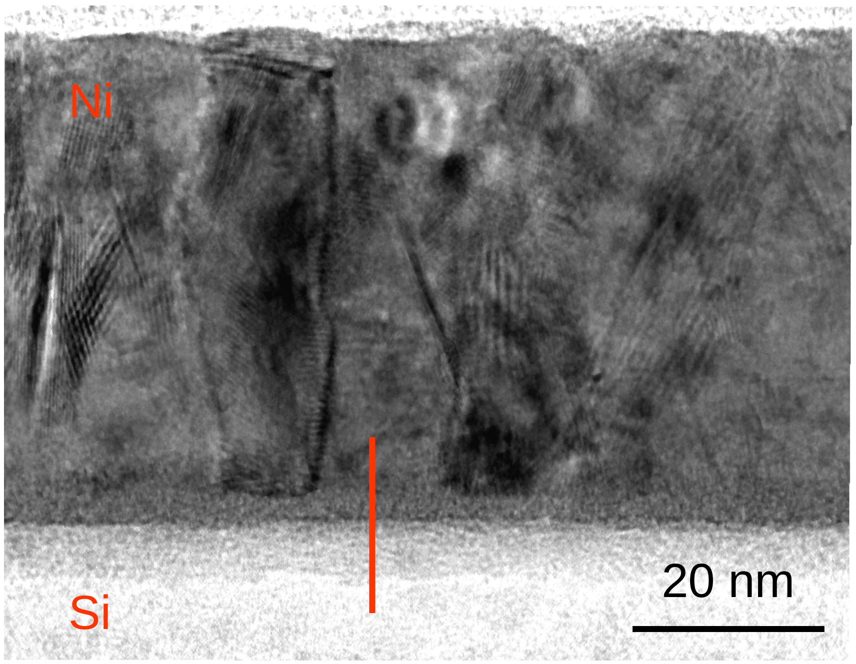
t (nm)	δ_s (nm)	δ_i (nm)
800	~2	« 0.01
200	~2	« 0.01
50	~2	~ 0.1



The values of M measured in our AFAM experiments were **not** influenced by the substrate!

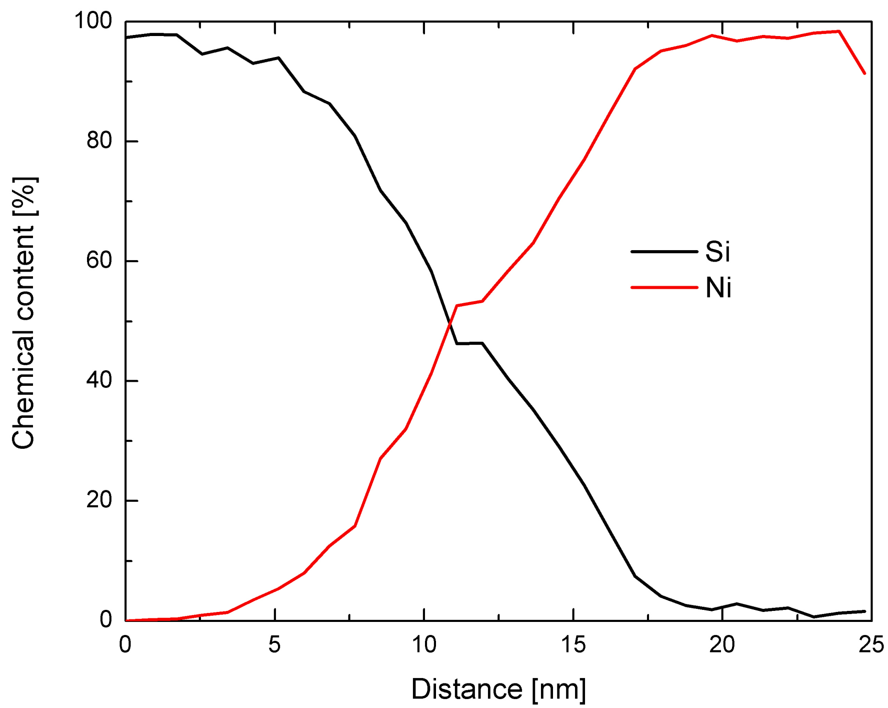
What about closed porosity (voids) and impurities?

Neither SEM or TEM images showed closed porosity (voids)



line scan

Analysis of the EDS spectrum from the sample showed only Si and Ni and no other significant elemental impurities



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Grain size effects?

	Ni_{800nm}	Ni_{200nm}	Ni_{50nm}
Grain size:	27 ± 10 nm	22 ± 5 nm	10 ± 3 nm

Polycrystalline materials consist of crystalline and intercrystalline (grain boundaries, triple junctions) components.

Crystalline component:	89 %	87 %	73 %

For nanocrystalline materials the effective elastic properties change with decreasing crystalline component

M_{eff}:	228 GPa	223 GPa	202 GPa
M_{AFAM} :	223 ± 28 GPa	220 ± 19 GPa	210 ± 26 GPa

The reduction in M observed in our experiments is caused by the increasing volume of the intercrystalline components.

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Summary

We measured the elastic properties of ultra-thin nickel film samples with AFAM.

The values of M we obtained were lower than those expected for a textured material.

Possible causes for the observed reduction in M:

The substrate influence on the measured values: NO

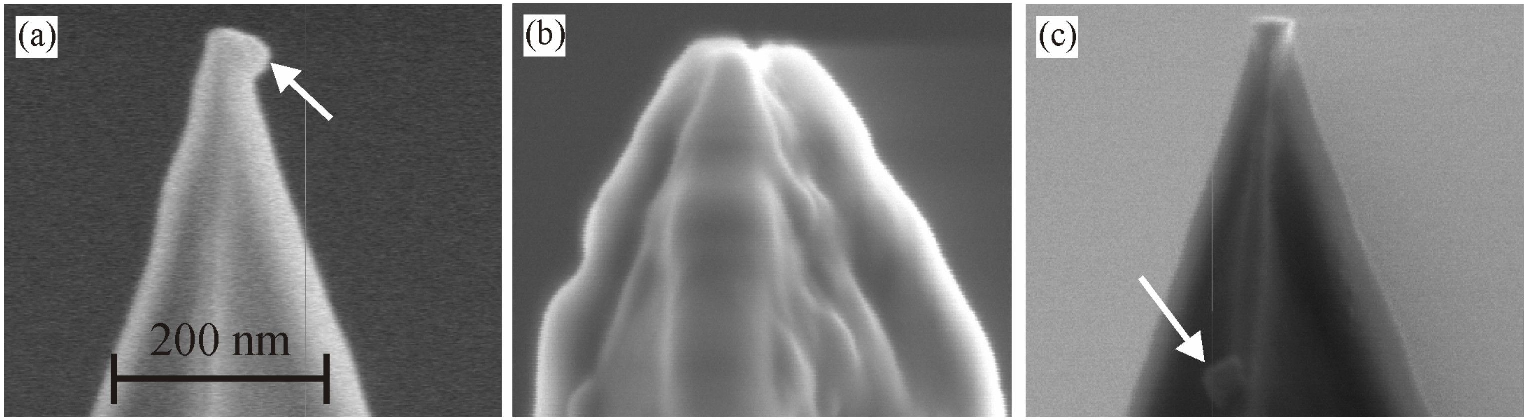
Presence of porosity, impurities, …. : NO

Grain size effect: YES

AFAM offers the flexibility to measure very thin films. The minimum thickness depends on the relative elastic properties, tip radius and applied loads.

Poster session, Wednesday evening, December 1

Influence of tip wear on atomic force acoustic microscopy experiments O10.16



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